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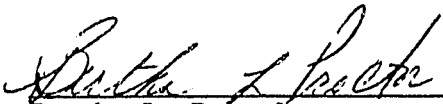
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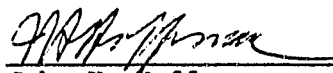
FINAL REPORT  
FOR  
STUDY OF OUTGASSING AND DECOMPOSITION  
OF SPACE SHUTTLE HEAT PROTECTION TILES, FILLERS, AND ADHESIVE

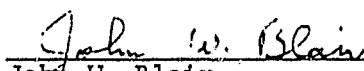
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COMPOUNDS DESORBING FROM THE SPACE SHUTTLE TILES

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The purpose of this project was to determine the chemicals desorbing from the space shuttle heat protection tiles. The original protocol for this project involved direct insertion probe mass spectrometry (DIPMS) analysis of the outgassing products from the tiles. This procedure allowed us to examine the desorbing chemicals for the tile material subjected to temperatures ranging between ambient temperature to 500°C at a pressure of  $10^{-3}$  torr. However, this method proved unsatisfactory due to the large number of compounds desorbing from the tiles. Analysis by DIPMS does not allow for separation of compounds with the same volatility (1). The large number of compounds desorbing from the tile material resulted in unresolved complex organic mixture.

A purge and trap technique (2) was then employed to collect and separate the chemicals desorbing from the tiles. The maximum temperature in this analysis was 280°C which is the gas chromatograph fused silica capillary column's temperature limit. The desorption was also carried out at atmospheric pressure with helium as the purge gas. A description of the modified protocol is given below. All compounds are tentatively identified and have not been confirmed.

## Material and Methods

### Sample Collection

Interior Tile Material: A tared, preconditioned (1 hour at 550°C) quartz tube (3 mm I.D. x 5 cm) was inserted directly into the tiles to a depth ranging between 3-4.5 cm. The

black protective skin on the surface of the tiles was removed prior to insertion of the quartz sample tube. Care was also taken to avoid the adhesive material on the bottom of the tiles. Samples represented a vertical profile of the upper half of most tiles tested. Aliquots of the tile material ranged between 0.8-1.1 grams. After collection of tile material the samples were placed in the purge and trap chamber for analysis.

**Exterior Tile Materials:** The black protective skin (0.3-0.5 mm thick) or the surface of the tiles was carefully removed and quantitatively transferred to the quartz desorption tubes. Aliquots of this material weighed between 0.3-0.45 grams. It should be noted that it was virtually impossible to separate all of the black skin from the white subsurface tile material.

#### Method

##### **Purge:**

The quartz tube containing the tile material was heated to 280°C and held at that temperature for 15 minutes. There was a constant flow of helium through the purge chamber to sweep the desorbing

compounds into the gas chromatograph where they were cryogenically trapped onto the fused silica capillary column. The inlet of the column was held at  $-30^{\circ}\text{C}$  throughout the desorption phase. After desorption the purge chamber was cooled to room temperature and the trapped compounds separated and analyzed by gas chromatography-mass spectrometry (GC-MS).

Instrumentation and Conditions;

Mass Spectrometer	Nermag R-10-10
Conditions	Electron Impact Mode
	Filiment current 70eV
	Mass Range 60-500 amu
	Scan Rate 2 msec per amu
Gas Chromatograph:	Carlo Erba (model 4160) equipped with a purge and trap unit constructed at UT-Dallas.
Conditions	Column: 30 meter-bonded fused silica capillary column DB5 (J&W)
	Carrier Gas: He.
Temperature Program:	The initial column temperature of $0^{\circ}\text{C}$ was maintained for one minute. The oven temperature was then raised at a rate of $5^{\circ}\text{C}$ per minute to a final temperature of $280^{\circ}\text{C}$ . The final temperature was maintained for 15 minutes.